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LUBRICATING OIL COMPOSITION FOR METAL PLASTIC WORKING  
[Kinzoku sosei kako yo junkatsu yu soseibutsu]

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APPLICANT(S)	(71):	NIPPON STEEL CORP.
TITLE	(54):	LUBRICATING OIL COMPOSITION FOR METAL PLASTIC WORKING
FOREIGN TITLE	[54A]:	KINZOKU SOSEI KAKO YO JUNKATSU YU SOSEIBUTSU

## 1. Title of the Invention

Lubricating Oil Composition for Metal Plastic Working

## 2. Claims

1. A lubricating oil composition for metal plastic working comprising a mixture of one kind out of a saturated fatty acid, unsaturated fatty acid, oxy fatty acid, and iso fatty acid and one or more kinds out of a mono, di, tri, or tetraester obtained by a reaction with a pentaerythritol.

2. The lubricating oil composition for metal plastic working cited in Claim 1 comprised by adding one kind out of a cationic surfactant, anionic surfactant, nonionic surfactant, phosphoric acid ester-based activator, phenol-based antioxidant, amine-based antioxidant, and boron-based antioxidant.

3. The lubricating oil composition for metal plastic working cited in Claim 1 comprised by adding a composition belonging to one or more groups out of compositions belonging to the respective groups (a), (b), and (c) below.

(a) Mineral oil, polyalkylene, microcrystalline wax.

(b) Japan tallow, carnauba wax, beeswax, rice bran wax.

(c) A diester of fat or oil, aromatic dibasic acid, and aliphatic alcohol; diester of aliphatic dibasic acid and aliphatic alcohol; ester of aromatic phenol and phosphoric acid; ester of aliphatic

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\*Numbers in the margin indicate pagination in the foreign text.

alcohol and phosphoric acid; ester of sperm oil or its hydrogenation product, an oxy acid, and an aliphatic alcohol; and partial ester of a polyester plasticizer, a polyalkylene glycol, a polyacrylic acid, and its alcohol.

4. The lubricating oil composition for metal plastic working cited in Claim 1 comprised by adding one kind out of a cationic surfactant, anionic surfactant, nonionic surfactant, phosphoric acid ester-based activator, phenol-based antioxidant, amine-based antioxidant, and boron-based antioxidant, and a composition belonging to one or more groups out of the composition belonging to one or more groups out of compositions [Translator's note: stated twice--see corrected Claim(s) in Amendments] belonging to groups (a), (b), and (c) below.

(a) Mineral oil, polyalkylene, microcrystalline wax.

(b) Japan tallow, carnauba wax, beeswax, rice bran wax.

(c) A diester of fat or oil, aromatic dibasic acid, and aliphatic alcohol; diester of aliphatic dibasic acid and aliphatic alcohol; ester of aromatic phenol and phosphoric acid; ester of aliphatic alcohol and phosphoric acid; ester of sperm oil or its hydrogenation product, an oxy acid, and an aliphatic alcohol; and partial ester of a polyester plasticizer, a polyalkylene glycol, a polyacrylic acid, and its alcohol.

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3. Detailed Specifications

The present invention relates to a lubricating oil composition for metal plastic working whereby an ideal lubrication effect is obtained by using it during plastic working of metals, such as drawing and ironing, and pressing.

Lubricating oils composed mainly of mineral oils or animal and vegetable oils and fats are applied in a practical manner in the above-mentioned metal plastic working fields. Although an effect was brought about per se, there was an inherent problem because it could not be manifested enough relative to various factors, such as lubrication properties and degreasing properties.

In view of these problems, the present invention intends to provide a lubricating oil composition for metal plastic working wherein the lubrication properties, oil stain resistance, rust preventability, emulsion dispersibility, and the like are far superior than those of conventional lubricating oil compositions for metal plastic working, while drawbacks, such as poor lubrication properties and poor degreasing properties, and the occurrence of galling flaws observed in conventional lubricating oil composition for metal plastic working are eliminated, the moldability is outstanding, it is excellent even as a post-pickling surface oil for steel sheets or the like, and also, the occurrence of grazing is suppressed and a high lubrication performance can be maintained, being characterized in that:

(1) A lubricating oil composition for metal plastic working comprising a mixture of one kind out of a saturated fatty acid, unsaturated fatty acid, oxy fatty acid, and iso fatty acid and one or more kinds out of a mono, di, tri, or tetraester obtained by a reaction with a pentaerythritol.

(2) A composition comprised by adding to the composition cited in above-mentioned (1) one kind out of a cationic surfactant, anionic surfactant, nonionic surfactant, phosphoric acid ester-based activator, phenol-based antioxidant, amine-based antioxidant, and boron-based antioxidant.

(3) A composition comprised by adding to the composition cited in above-mentioned item (1) a composition belonging to one or more kinds out of compositions belonging to groups (a), (b), and (c) below.

(4) A composition comprised by adding to the composition cited in above-mentioned item (2) a composition belonging to one or more groups out of compositions belonging to groups (a), (b), and (c) below.

(List)

(a) Mineral oil, polyalkylene, microcrystalline wax.

(b) Japan tallow, carnauba wax, beeswax, rice bran wax.

(c) A diester of fat or oil, aromatic dibasic acid, and aliphatic alcohol; diester of aliphatic dibasic acid and aliphatic alcohol; ester of aromatic phenol and phosphoric acid; ester of aliphatic alcohol and phosphoric acid; ester of sperm oil or its hydrogenation product, an oxy acid, and an aliphatic alcohol; and partial ester of a

polyester plasticizer, a polyalkylene glycol, a polyacrylic acid, and its alcohol.

Furthermore, the lubricating oil composition for metal plastic working of the present invention is not only a composition per se, it is also exceedingly effective in cases where it is used as an emulsion dispersed in water. For example, it is not only outstanding as a coolant and lubricant during a metal working operation, but it also has an effect of remarkably reducing a friction force by providing the above-mentioned composition emulsion on punch and die faces during drawing and ironing work or on the contact faces between the rolling rolls and steel sheets during cold rolling.

As a result of a variety of research, the inventors discovered that the biggest contribution—the excellent lubrication properties—in the present invention was due to the pentaerythritol fatty acid ester contained in the composition of the present invention. That is to say, they discovered that the partial ester of pentaerythritol and fatty acid and the unsaturated ester copresent therewith had a unique structure in which the former had an OH group and ester group copresent in the molecule, or four ester groups coexisted in one molecule and the arrangement of these was a tetrapod shape. Thus, the attracting force to a metal surface was strong, and sliding lubrication was excellent. Moreover, the pentaerythritol ester had a lower melting point than fats and oils comprising the same fatty acids, and therefore, it was outstanding as a press oil and as a post-

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pickling surface oil, and the surface activity was better than that of fats and oils; hence, uniform coating thereof on a steel sheet was easy, it could be used in an emulsion state at low temperatures, and due to the presence of an OH group exhibiting hydrophilicity, it was suitable for a working lubricant having outstanding emulsion dispersibility and good cooling properties.

The fatty acids forming the pentaerythritol partial ester and the saturated ester include fatty acids comprising beef tallow, lard, lanolin, canola oil, sunflower oil, and their hydrogenated products; saturated or unsaturated fatty acids, such as caprylic acid, palmitic acid, stearic acid, and oleic acid; naphthenic acid; iso fatty acids obtained in an oxo method or the like; etc. The ester substituent is a mixture of mono, di, and tri partial esters and a compound in which four OHs were esterified with the mixture.

Although only a mixture in which the partial ester of pentaerythritol and a fatty acid and a tetraester that were mixed can be used as a lubricant for plastic working, and depending on the die and punch surface temperature during plastic working, the amount of plastic deformation, the deformation speed, the overhang, and the proportion of the throttling element. That is to say, depending on this compounding, the lubrication properties are improved due to actions, such as compacting of the compounded adhesive film of pentaerythritol ester on the metal surface and regulating the fusing of the lubricant in the plastic working region, the properties of the



lubricating oil film can be adjusted by compounding a composition belonging to one or more kinds out of a composition belonging to each group including (a) a mineral acid, (b) Japan tallow, carnauba wax, beeswax, rice bran wax, and a polymer compound, such as polybutene and polypropylene, and (c) a diester of fat or oil, aromatic dibasic acid, and aliphatic alcohol; diester of aliphatic dibasic acid and aliphatic alcohol; ester of aromatic phenol or aliphatic alcohol and phosphoric acid; ester of sperm oil and its hydrogenation product, oxy acid, and aliphatic alcohol; and partial ester of a polyester plasticizer, a polyalkylene glycol, a polyacrylic acid, and its alcohol.

Moreover, the HLB (hydrophilic-lipophilic balance) of a mixture of the pentaerythritol partial ester and tetraester changes depending on the chain length of the fatty acid constituents and the unbonded OH groups, and the emulsion dispersibility in water may be adjusted.

For example, although the emulsion dispersibility of the composition in which a monoester, diester, and triester were mixed at a 1:1:1 ratio with a pentaerythritol beef tallow fatty acid is extremely good, the emulsion dispersibility and the degreasing properties may not be sufficient when compounded with a mineral oil, Japan tallow, carnauba wax, beeswax, bran oil wax, fat or oil, polybutene, polypropylene, sperm oil hydrogenated product esterified with a dibasic acid, polyester plasticizer, etc.

In order to additionally improve this, in this case it is desirable to add a nonionic surfactant, anionic surfactant, cationic

surfactant, or the like to a composition containing the above-mentioned pentaerythritol ester. In particular, when it is necessary to degrease steel sheets after plastic working, such as press working, in a short time, it is essential to compound the above-mentioned surfactant with the above-mentioned composition containing the pentaerythritol ester in advance.

Next, when a lubricating composition containing a fatty acid, aliphatic alcohol, or the like is exposed to high humidity after plastic working, an oil stain is produced on the plastic worked surface.

According to the research of the inventors, it was discovered that the primary factor that this oil stain occurred included, in addition to the extrinsic temperature and humidity factors and the intrinsic factor of the oxide film composition on the surface of the steel sheets, a factor that the lengths of the double bonds of the coating composition, the free carboxylic acids, OH groups, ester groups, and alkyl groups are important, and also, they surmised that the composition of this oil stain was composed mainly of  $\text{FeOOH}$  due to the steel sheets. As a result of researching a method for preventing this oil stain, the inventors discovered that if the compounds described below are compounded with the composition of the present invention, the occurrence of oil stains could be completely prevented even during long-term preservation, and in high-temperature and high humidity environments.

That is to say, paranitrophenol, zinc dialkyl dithiophosphate [Translator's note: misspelled in original],  $\alpha$ -naphthyl amine, Naphthol, paranitrobenzoic acid,  $\alpha$ -naphthyl amine, diphenyl amine, paraaminophenol, paraphenylene diamine, diphenyl phenylene diamine, dinaphthyl paraphenylene diamine, tetramethyl diaminodiphenyl methane, ditert-butyl paracresol, ditert-amyl iodoquinone, ditert-butyl hydroquinone, butylidene bis-tert-butyl meta-cresol, alkyldiallyl phosphite, a boroxalophenanthrene derivative, etc. If desired, small amounts of other kinds of additives can be compounded with the composition of the present invention. That is to say, known rust preventives, preservatives, defoaming agents, pour point depressants, and the like are cited for these additives.

Compounding of the respective constituents can be performed ideally by heating and mixing them.

A temperature at which the respective constituents dissolve and moreover do not decompose, and specifically a range of from 60 to 150°C should be adopted for the temperature in this case. The mixing system may be any given known system in which the respective constituents can be homogeneously mixed. When the lubricating oil composition for metal plastic working of the present invention is used in an emulsion form, the respective constituents can be completely homogeneously heated and mixed in the above-mentioned mixing method, and moreover, some of the constituents may be added in an aqueous phase when they are in an emulsion form.

An excellent lubrication performance, emulsion dispersibility, rust prevention, oil stain resistance, and degreasability are manifested on the basis of the respective features of the respective compounded constituents and their synergisms. In particular, when the present invention is used as an oil for cold rolling steel for example, more extremely satisfactory results, wherein the rolling performance thereof is improved 20% or more than that of palm oil, are obtained.

Practical examples of the lubricating oil composition for metal plastic working in the present invention are shown next in Table 1.

(The numbers denote the mixing rates (% by weight) unless otherwise noted.)

Table 1

配合組成		1	2	3	4	5	6	7	8	9	10	11	12	13
ベンゾエリス トリオール 牛脂脂肪酸 エステル トリオステル デトラステル	モノエステル	55	40	20	10	20	15	15	15	20	20	20	76	牛 脂 系 市 販 基 油 ( 精 化 値 170
	ジ エステル	55	30	40	50	40	50	50	50	40	40	40		
	トリエステル	54	20	30	20	30	30	30	30	30	30	30		
	デトラステル	0	10	10	10	10	5	5	5	10	10	10		
牛脂 (エキストラファイン)			90	84.5	68.5		30	30	30	39.5	19	29	25	
鉱油 (粘度指数 65)						78	48	48	48	30	30			
ホ 素							10		5	3				
カルナウバ 脂								10	5					
ポリエチレン(コ-モノ)ラウリル エーテル (H, L, B=10)				3		3	3	3	3	3				
アルキルアミン エチレンオキシド 酸性脂肪酸エステル (H, L, B=7.5)					0.6	3	3	3	3	3				
モノエタノールアミン					0.1	0.5	0.5	0.5	0.5	0.5				
4,6ジメチルシクロヘキサン				0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2		
2,4,6-トリメチルフェノール				0.1	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2		
清 油 系				0.1	0.1	0.05	0.05	0.05	0.05	0.5	0.5	0.5		
防 腐 剤				0.1	0.1	0.05	0.05	0.05	0.05	0.1	0.1	0.1		



Rolling machine: double rolling machine; work roll: 200 mm×250 mm diameter

Rolling speed: 16 m/sec

Rolling reduction ratio: about 30%

Emulsion lubricating liquid:

Concentration: 3 to 5% by weight

Temperature: 40°C

(Provided the concentration of the palm oil was 20% by weight and tested at a temperature of 80°C.)

Emulsion lubricating liquid supply system:

spray-coated through roll inlet,

spray pressure: 1.5 to 2.0 kg/cm<sup>2</sup>

Rolling index (RI):

Found by rolling a steel sheet through a predetermined roll gap, [illegible] the coefficient of extension of a standard oil and the coefficient of extension of the sample oil.

$$RI = \frac{L - 300}{L_0 - 300}$$

L: distance (mm) between steel strip gage marks after rolling with sample oil

L<sub>0</sub>: distance (mm) between steel strip gage marks after rolling with standard oil

Provided the distance between steel strip gage marks before rolling was set to 300 mm. The emulsification stability is tested by

charging 400 mL of the emulsion of the present invention from an emulsion tank through a separatory funnel during the above-mentioned cold rolling and letting it stand for 30 minutes in a constant-temperature tank at the same temperature (40°C). After letting it stand punctually for 30 minutes, it is divided into a 100 mL flask, and the amounts of oil in the flask at the start and at the end are measured.

$$\text{Emulsion stability index (ESI)} = (\text{Amount of oil in 100 mL flask at the start}) / (\text{Amount of oil in 100 mL flask at the end})$$

The emulsion stability was tested according to the emulsion stability index (ESI).

The test results for the rolling performance and emulsion stability of the respective lubricating [Translator's note: misspelled in source-see Amend. (2)] oil compositions in Table 1 are shown in Table 2.

Table 2

性 能 潤滑油組成物	R I	E S I
1	1.25	1.00
2	1.15	0.60
3	1.12	0.85
4	1.10	0.85
5	0.96	0.90
6	0.98	0.90
7	0.98	0.90
8	0.98	0.90
9	0.99	0.95
10	1.05	0.90
11	1.18	0.90
12 パーA油	1.10	0.80
13 牛脂系市販油	0.93	0.50

[Translator's note: please refer to the original description]

Key:

Lubricating Oil Composition No.	Performance	
12 Palm Oil		
13 Beef tallow-based commercially-available oil		

It is seen from Table 2 that the composition of the present invention has extremely outstanding performance as a lubricant for cold rolling.

Namely, the rolling performance of the composition nos. 1, 2, 3 and 11 in which no mineral oil was compounded is about 10 to 25% more outstanding than palm oil, and even the composition nos. 5 to 10 in which a mineral oil was compounded exhibit rolling lubrication properties to nearly equivalent to those of palm oil. Moreover, it is seen from the ESI value that the emulsion stability derived from the chemical structure of pentaerythritol fatty acid ester of the lubricating oil composition emulsion in this practical example is excellent.

#### <Practical Example 2>

Low-carbon steel strips obtained by the cold rolling in Practical Example 1 were electrolytically degreased and then annealed.



The treatment [Translator's note: misspelled in original--see Amend. (3)] time for the electrolytic degreasing was curtailed, and a normally sufficient degreasing for difficult-to-obtain conditions was chosen. The proportion of the electrolytically-degreased low-carbon steel strips that was wetted with water was judged with the unaided eye.

Moreover, low-carbon steel that was not electrolytically degreased was annealed and the state of occurrence of oils stains after the annealing was judged with the unaided eye.

The test results for the electrolytic degreasing properties and oil stains are shown in Table 3.

As is evident from Table 3, the electrolytic degreasing properties of the composition of the present invention are more outstanding than those of the palm oil and beef tallow-based commercially-available oil. However, since the adsorption properties of the functional groups of the pentaerythritol fatty acid ester alone are good, as described above, the degreasing performance is slightly /6 inferior.

Moreover, it is seen that although slight, the oil is more outstanding for oil stains than the palm oil and the beef tallow-based commercially-available oil. The cold-rolled low-carbon steel strips are wound in a coiled state as is at 35°C and an 80% RH atmosphere for about 2 weeks. As a result of opening and observing the coil, there was not generation of rust owing to oil stains.

Table 3

組成物番号 \ 性能	水ぬれ (%)	オイルステーン (%)
1	50~70	痕 跡
3	80~100	痕 跡
4	70~90	な し
5	90~100	な し
8	90~100	な し
12 パーム油	60~80	10~15
13 牛脂系市販油	70~90	2~5

[Translator's note: please refer to the original description]

Key:

Composition No. \ Performance	Water Wetting (%)	Oil Stain (%)
		Stained
		Stained
		No stains
		No stains
		No stains
12 Palm oil		
13 Beef tallow-based commercially-available oil		

### <Practical Example 3>

The drawing and ironing [Translator's note: English misspelled in original--see Amend. (4)] workability was examined in the following method.

Sample: matte material, plating: #75/#50, unmelted thickness: 0.34 mm

Molding conditions: speed 1.8 m/sec

成形段階 \	1st Ironing	2nd	3rd	4th	
ダイス径	20.55	20.40	20.30	20.20	
肉厚(90°)	0.275	0.20	0.15	0.10	Total
減面率	19.1	27.3	25.0	33.3	70.6

[Translator's note: please refer to the original description; English 2<sup>nd</sup> and 3<sup>rd</sup> misspelled in original--see Amend. (5)]

Key:

Molding Stage				
Die diameter				
Thickness (clearance)				
Area reduction rate				

Lubrication conditions: temperature 40°C; a 10% by weight emulsion was sufficiently spray-coated at each stage

Moldability evaluation: molding load, molded height, and fracture characteristics were examined. Moreover, the fracture characteristics were denoted by  $n_2/n_1$  (molding work was performed on ten samples ( $n_1$ ) and the number of samples that fractured after molding was defined by  $n_2$ ).

The test results of the drawing and ironing work [Translator's note: English misspelled in original--see Amend. (4)] are shown in Table 4.

Table 4

潤滑剤組成物	成形段階	破断特性	成形高さ	成形荷重
No. 3	1 <sup>st</sup> Ironing	10/10	24mm	0.70 <sup>ton</sup>
	2 <sup>nd</sup>	10/10	32	0.45
	3 <sup>rd</sup>	10/10	42	0.40
	4 <sup>th</sup>	8/10	55	0.40
No. 5	1 <sup>st</sup>	10/10	22	0.85
	2 <sup>nd</sup>	10/10	30	0.60
	3 <sup>rd</sup>	10/10	40	0.55
	4 <sup>th</sup>	6/10	50	0.58
No. 8	1 <sup>st</sup>	10/10	24	0.75
	2 <sup>nd</sup>	10/10	33	0.50
	3 <sup>rd</sup>	10/10	43	0.45
	4 <sup>th</sup>	8/10	56	0.45
市販鉱物油 DQI油 { 粘度 50.4 <sup>cst</sup> (38°C) 凝結点 -17°C 比重 0.909	1 <sup>st</sup>	10/10	22	0.92
	2 <sup>nd</sup>	10/10	30	0.69
	3 <sup>rd</sup>	8/10	40	0.62
	4 <sup>th</sup>	7/10	50	0.66

[Translator's note: please refer to the original description]

Key:

Lubricant Composition	Molding Stage	Fracture Characteristics	Molding Height	Molding Load
Commercially-available mineral oil DQI Oil Viscosity: 50.4 cst (38°C) Pour point: 17°C Specific gravity: 0.909				

As is evident from Table 4, excellent characteristics were shown for a lubricant for drawing and ironing [Translator's note: English and Japanese misspelled in original--see Amends. (4, 6)] work, and also, the appearance of the inner and outer surfaces of the molded can were in particular more outstanding than that for a commercially-available oil, and the occurrence of galling flaws was extremely insignificant. Thus, it has been demonstrated that the lubricating oil composition of the present invention has an excellent lubrication action from the standpoint of working.

7. Inventors (see title page)

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Procedural Amendment

May 22, 1974

Dear Hideo Saito, Commissioner of the JPO

1. Designation of Case

Patent Application No. 48-58720

2. Title of the Invention Lubricating Oil Composition for Metal

Plastic Working

3. Party Affecting the Amendment

Connection with Case      Applicant

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5. Date of Effect of Amendments      Voluntary

6. Objects of Invention              Claims and Detailed Specifications  
columns of Specification

7. Amendment Details              As per enclosure

[stamp: May 22 [illegible]]

- (1) Amend the Claims as per enclosure.
- (2) Correct "respective 'lubrication oil' compositions" misspelling on second line from bottom of page 15 of the Specification.
- (3) Correct misspelling of "treatment time" on line 9, page 17 of the Specification.
- (4) Correct English misspellings of "Drawing & Ironing" on line 3, page 19, second line from bottom of page 19, and line 2, page 21.
- (5) Correct English misspelling of 2<sup>nd</sup> and 3<sup>rd</sup> in table on line 6, page 19.
- (6) Correct misspelling of "rolling" on line 2, page 21.

Claim(s)

1. A lubricating oil composition for metal plastic working comprising a mixture of one kind out of a saturated fatty acid, unsaturated fatty acid, oxy fatty acid, and iso fatty acid and one or more kinds out of a mono, di, tri, or tetraester obtained by a reaction with a pentaerythritol.

2. The lubricating oil composition for metal plastic working cited in Claim 1 comprised by adding one kind out of a cationic surfactant, anionic surfactant, nonionic surfactant, phosphoric acid ester-based activator, phenol-based antioxidant, amine-based antioxidant, and boron-based antioxidant.

3. The lubricating oil composition for metal plastic working cited in Claim 1 comprised by adding a composition belonging to one or

more groups out of compositions belonging to the respective groups (a), (b), and (c) below.

(a) Mineral oil, polyalkylene, microcrystalline wax.

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(b) Japan tallow, carnauba wax, beeswax, rice bran wax.

(c) A diester of fat or oil, aromatic dibasic acid, and aliphatic alcohol; diester of aliphatic dibasic acid and aliphatic alcohol; ester of aromatic phenol and phosphoric acid; ester of aliphatic alcohol and phosphoric acid; ester of sperm oil or its hydrogenation product, an oxy acid, and an aliphatic alcohol; and partial ester of a polyester plasticizer, a polyalkylene glycol, a polyacrylic acid, and its alcohol.

4. The lubricating oil composition for metal plastic working cited in Claim 1 comprised by adding one kind out of a cationic surfactant, anionic surfactant, nonionic surfactant, phosphoric acid ester-based activator, phenol-based antioxidant, amine-based antioxidant, and boron-based antioxidant, and a composition belonging to one or more groups out of the compositions belonging to groups (a), (b), and (c) below.

(a) Mineral oil, polyalkylene, microcrystalline wax.

(b) Japan tallow, carnauba wax, beeswax, rice bran wax.

(c) A diester of fat or oil, aromatic dibasic acid, and aliphatic alcohol; diester of aliphatic dibasic acid and aliphatic alcohol; ester of aromatic phenol and phosphoric acid; ester of aliphatic alcohol and phosphoric acid; ester of sperm oil or its hydrogenation



product, an oxy acid, and an aliphatic alcohol; and partial ester of a polyester plasticizer, a polyalkylene glycol, a polyacrylic acid, and its alcohol.)